National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 861

Nickel-based Superalloy

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of techniques employed in the determination of sulfur and phosphorus in nickel-based alloys. SRM 861 consists of a glass jar containing 50 g of metal chips that have passed through a 600 µm (30 mesh) screen.

The certified value for sulfur is listed below and the reference value for phosphorus is listed on the next page. For both elements, the values are reported as mass fractions [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for chemical measurement of reference materials [2], and uncertainties are assessed according to the ISO Guide [3].

Certified Sulfur Value and Uncertainty: The certified value for sulfur is based on a single NIST primary method, isotope dilution thermal ionization mass spectrometry [4-6]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [2].

Certified Value of Sulfur: 0.561 mg/kg ± 0.078 mg/kg

The uncertainty in the certified value for sulfur is expressed as an expanded uncertainty, U, using the methods described in the ISO Guide [3]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with measurement uncertainty and sulfur inhomogeneity; k is a coverage factor used to control the confidence level of the expanded uncertainty. The coverage factor, $k = 2.23$, is determined from the Student's t-distribution with 10 degrees of freedom and corresponds to an approximate 95 % confidence interval.

Expiration of Certification: The certification of SRM 861 is valid, within the measurement uncertainties specified, until **30 January 2015** provided the SRM is handled in accordance with the instructions given in this certificate (see Instructions for Use). This certification is nullified if the SRM is damaged, contaminated, or modified in any way other than its intended use.

The coordination of the technical measurements leading to certification was performed by W.R. Kelly of the NIST Analytical Chemistry Division. Homogeneity testing of the chipped material was performed by R. Kunish of Howmet Corporation, Whitehall, MI.

Isotope dilution thermal ionization mass spectrometry analyses were performed by W.R. Kelly, J.L. Mann, and R.D. Vocke of the NIST Analytical Chemistry Division. Radiochemical neutron activation analysis was performed by R.L. Paul of the NIST Analytical Chemistry Division.

Statistical analyses leading to establishment of the certified and reference values were performed by W.F. Guthrie of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by N.M. Trahey.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 28 March 2001

Nancy M. Trahey, Chief
Standard Reference Materials Program

Reference Phosphorus Value and Uncertainty: The reference value for phosphorus is based on a new radiochemical neutron activation analysis method developed at NIST [7-8]. A NIST reference value is a noncertified value and is the best estimate of the true value. The value does not meet NIST criteria for certification and is provided with an associated uncertainty that may not include all sources of uncertainty [2].

Reference Value of Phosphorus: 12.71 mg/kg ± 0.71 mg/kg

The uncertainty in the reference value for phosphorus is expressed as an expanded uncertainty, *U*, using the methods described in the ISO Guide [3]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effect of uncertainty components associated with measurement precision, and k is a coverage factor used to control the confidence level of the expanded uncertainty. The coverage factor, $k = 2$, is determined from the normal distribution.

INSTRUCTIONS FOR USE

Sampling: The unit should be thoroughly mixed by rotating the bottle before sampling. A minimum sample mass of 100 mg should be used for analytical determinations to be related to the sulfur and phosphorus values provided in this certificate. It is recommended that when not in use, each unit of SRM 861 be stored tightly capped and isolated from the atmosphere to prevent contamination.

PREPARATION, HOMOGENEITY, AND ANALYSIS

Source and Preparation of Material: The parent material for this SRM was purchased from Howmet Corporation and is a superalloy of a type that is commonly used to fabricate single-crystal high temperature turbine blades for jet aircraft engines. The original material was in the form of two bars (9 cm in diameter by 122 cm in length), each weighing about 45 kg. The bars were dry chipped at Analytical Reference Materials International (ARMI), Evergreen, CO, and the chips placed in 55 glass jars. Before blending the chips, two samples were taken from each jar and the sulfur determined by a combustion technique at Howmet Corporation under the direction of R. Kunish to check for the possibility of inadvertent contamination during the chipping process. All jars had essentially the same sulfur concentration. The blending of the contents of the 55 jars for distribution into SRM units was performed at NIST under the direction of D.G. Friend of the NIST Standard Reference Materials Program.

REFERENCES

[1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).

[2] May, W.E., et al., "Definitions of Terms and Modes used at NIST for Value-Assignment of Reference Materials for Chemical Measurement," NIST Special Publication 260-136, U.S. Government Printing Office, Washington DC, (2000).

[3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); available at http://physics.nist.gov/Pubs/.

[4] Paulsen, P.J. and Kelly, W.R., "Determination of Sulfur as Arsenic Mono-sulfide Ion by Isotope Dilution Thermal Ionization Mass Spectrometry," Anal. Chem., **56**, pp. 708-713, (1984).

[5] Kelly, W.R. and Paulsen, P.J., "Precise and Accurate Determination of High Concentrations of Sulfur by Isotope Dilution Thermal Ionization Mass Spectrometry," Talanta, **31**, pp. 1063-1068, (1984).

[6] Kelly, W.R., Paulsen, P.J., Murphy, K.E., Vocke, R.D., and Chen, L.-T., "Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry," Anal. Chem., **66**, p. 2505, (1994).

[7] Paul, R.L., "Determination of Phosphorus in Steels by Radiochemical Neutron Activation Analysis," J. Radioanal. Nucl. Chem., **234**, pp. 55-58, (1998).

[8] Paul, R.L., "Measurement of Phosphorus in Metals by RNAA," J. Radioanal. Nucl. Chem., **245**, pp. 11-15, (2000).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.